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High-accuracy x-ray line standards in the 3-keV region

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A set of 14 high-accuracy x-ray transition energies in the 2.4–3.1 keV range is presented, which can be used as x-ray standards. They were measured in two- to four-electron sulfur, chlorine, and argon ions produced in an electron-cyclotron resonance ion source, using a single spherically bent crystal spectrometer. The results include the first measurement of six transitions and improve the accuracy of six other experimental values. These measurements considerably extend the set of high-accuracy x-ray energies reported for highly charged ions. Their relative uncertainties range from 1 to 10 ppm. Theory only reaches such a precision in one- and two-electron ions. Our results thus have two distinct applications. On the one hand, they test predictions in two-electron ions [Artemyev, Shabaev, Yerokhin, Plunien, and Soff, Phys. Rev. A **71**, 062104 (2005)], at the precision level of some two-photon QED contributions. We observe an agreement with theory for most of the transitions. On the other hand, the three- and four-electron ion transitions provide new benchmark energies for the calculation of missing theoretical contributions, such as Auger shifts or electronic correlations. Spectra were analyzed with an x-ray tracing simulation that contains all the relevant physics of the spectrometer.

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I. INTRODUCTION

The theoretical study of simple atomic systems, coupled with an ever-increasing accuracy in measurements, has led to numerous advances in physics. A famous example is the measurement of the Lamb shift in hydrogen [1] and its crucial contribution to the development of QED. A more recent example is the remarkable accuracy attained on the $1S$-$2S$ transition in hydrogen [2,3], which motivated the development of the frequency comb and strongly influences the values of multiple fundamental constants [4]. This article presents a set of energies measured with a high accuracy in two- to four-electron ions of sulfur, chlorine, and argon. They can be used both as high-accuracy x-ray standards and as benchmarks for future atomic calculations.

There is a growing need for reliable, precisely known x-ray standards for the characterization and calibration of instruments, for instance, at synchrotrons or x-ray free-electron laser facilities. The x-ray lines used for this purpose are traditionally produced with x-ray tubes or by fluorescence in suitable solid or gas targets. Many such x-ray lines have thus recently been critically evaluated and compiled [5]. Their energy is known to an accuracy of about 1 ppm in the best cases; below 5 keV, two reference transitions (found in medium Z elements, Z being the nuclear charge number) reach the part-per-million level [5], Table I.

However, using reference lines produced by medium-Z elements in x-ray tubes or by fluorescence suffers from a few drawbacks. Spectral lines are typically a superposition of numerous, broad component lines whose respective intensities generally depend on the experimental conditions. The several degrees of freedom involved in the line fits are thus enhancing the possibility of systematic errors. The line components include both satellite lines (which are typically created by shake-off) and the many transitions coming from the coupling of inner and valence shell holes (which in turn comes from the large number of electrons and from the open valence shells in the elements used—see, e.g., Refs. [6–9]). Individual component widths are furthermore significant because of large Auger transition probabilities (for example, the $K\alpha$ lines of argon have a width of 0.69 eV [10]). In addition, the energies of the components generally depend on both composition and contamination of the target (chemical shift) and on the excitation method and energy (see, e.g., Refs. [11,12]).

These limitations are largely overcome by using fewer-body systems like exotic atoms [13] and highly charged ions, which have thus been proposed as sources of x-ray standards [14,15]. An example is the $1s\,2s\ {}^3S_1 \rightarrow 1s^2\ {}^1S_0$ $M1$ transition in

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TABLE I. Experimental energies in electron volts of transitions of two- to four-electron ions. The left part of the table shows direct measurements, namely, the energy differences between the transitions of interest and the reference transitions ($M1$ lines). The right part of the table shows the absolute energy for transitions, calibrated using the theoretical energies for the reference transitions [21], reproduced in the last row. Uncertainties (standard deviations) on the last digits are indicated inside parentheses. Newly measured energies are in italics. Values improve previous measurements in most cases by a factor of 3 to 50. Larger uncertainties come from lower statistics.

Transition	S ($Z = 16$)	Cl ($Z = 17$)	Ar ($Z = 18$)	S ($Z = 16$)	Cl ($Z = 17$)	Ar ($Z = 18$)
	Direct measurements: Energy difference with the reference transition			Energy of the transition: Calculated values using the reference transition		
$1s\,2p\ ^1P_1 \rightarrow 1s^2\ ^1S_0$			35.419(11)	3139.567(11)		
$1s\,2p\ ^3P_2 \rightarrow 1s^2\ ^1S_0$	18.388(20)		22.143(11)	2448.739(20)		3126.291(11)
$1s\,2p\ ^3P_1 \rightarrow 1s^2\ ^1S_0$	16.798(9)		19.341(12)	2447.150(9)		3123.489(12)
$1s\,2s\,2p\ ^2P_{3/2} \rightarrow 1s^2\,2s\ ^2S_{1/2}$	*7.755(3)*	*8.813(3)*	9.974(2)	*2438.106(3)*	*2765.678(3)*	3114.122(2)
$1s\,2s\,2p\ ^2P_{1/2} \rightarrow 1s^2\,2s\ ^2S_{1/2}$	*6.764(5)*	*7.513(5)*	8.303(2)	*2437.115(5)*	*2764.378(5)*	3112.451(2)
$1s\,2s^2\,2p\ ^1P_1 \rightarrow 1s^2\,2s^2\ ^1S_0$	*−11.764(6)*	*−12.130(25)*	−12.372(3)	*2418.587(6)*	*2744.735(25)*	3091.776(3)
Reference transition [21]						
$1s\,2s\ ^3S_1 \rightarrow 1s^2\ ^1S_0$				2430.3512(3)	2756.8648(3)	3104.1483(4)

two-electron ions. Its energy was recently measured with a 2.3-ppm accuracy [16]. It is so narrow that it can also be used for the characterization of the response function of x-ray spectrometers [17]. A few additional high-accuracy x-ray transitions in medium-Z highly charged ions were recently measured: a 2-ppm measurement in He-like argon ($Z = 18$) [18], 10-ppm measurements in H-like chlorine ($Z = 17$) [18] and argon [19], and 10- and 15-ppm Lyman measurements in germanium ($Z = 32$) [20]. Such few-electron ions, besides providing useful experimental x-ray standards, are a particularly interesting testing ground for atomic calculation methods. Medium-Z ions also have the advantage of being sensitive both to relativistic and QED effects (which contribute more at high Z) and to interelectronic correlations (which are relatively more important for low-Z ions).

This article presents high-accuracy measurements of x-ray transitions observed in highly charged ions. Their energies lie in the 2.4–3.1 keV region; the accuracy achieved ranges from about 10 ppm down to 1 ppm. These energies can be found in Table I. The x-ray lines are produced by $n = 2$ to $n = 1$ transitions in two- to four-electron ions of sulfur ($Z = 16$), chlorine ($Z = 17$), and argon ($Z = 18$) (n is the principal quantum number). A typical spectrum is shown in Fig. 1. For two-electron ions, these results are precise enough to test theoretical calculations of the energy differences between excited levels. For ions with more electrons, our measurements are much more precise than theoretical predictions. Therefore, these measurements can be used as benchmarks for relativistic many-body calculations in three- and four-electron ions. A comparison between experiment and theory would test calculations of electronic correlation energies, which are often predicted with different values by different calculation methods [22], and of Auger shifts, for which to the best of our knowledge published results are limited to neutral atoms with a K, L, or M hole [5].

II. EXPERIMENTAL APPROACH

The type of spectrometer which was used is not suitable for absolute measurements of energies, but instead gives excellent measurements of energy differences between transitions. We chose the $1s\,2s\ ^3S_1 \rightarrow 1s^2\ ^1S_0$ $M1$ transition of two-electron ions as a reference (see Fig. 1); its reference energy was taken from the theoretical calculation of Ref. [21]. This transition is particularly suitable for parts-per-million-level measurements:

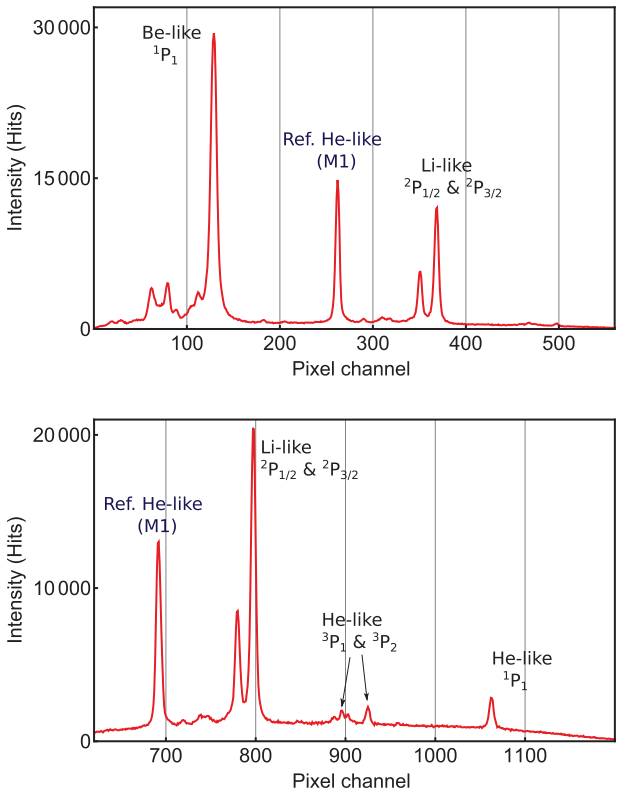

FIG. 1. (Color online) Spectra observed in argon ($Z = 18$) in about 1 h (each). Each grid spacing corresponds to approximately 10 eV (100 CCD pixels), the reference line ($M1$) being located here approximately at 3104 eV. The spectra for chlorine ($Z = 17$) and sulfur ($Z = 16$) are similar. The lines studied in this article are $n = 2 \rightarrow 1$ transitions; see Table I for more information. All the energies were measured relative to the He-like $M1$ calibration line. The spectral range of 60 eV was obtained on a single image thanks to the large x-ray source and detector.

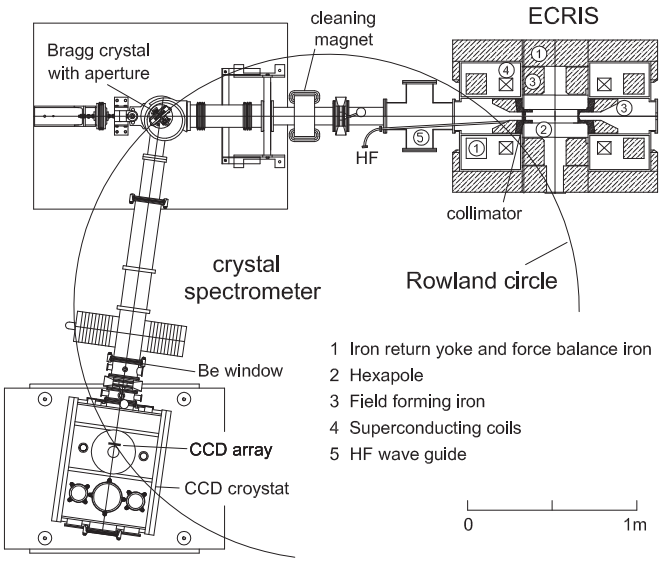

FIG. 2. Experimental arrangement of the ECRIS and crystal spectrometer (adapted from Ref. [17]). The source is connected through a vacuum tube to a crystal chamber, where a bent crystal reflects emitted x rays. The x rays are recorded with a two-dimensional position-sensitive detector made of charge-coupled devices (CCDs).

(i) its spectral line is bright in an electron-cyclotron resonance ion source (ECRIS) plasma; (ii) its energy is predicted to 0.1 ppm (about 0.4 meV) [21], and (iii) because of the long lifetime of the initial state (about 200 ns) [23], its natural width is negligibly small.

The ions were produced in a dedicated ECRIS [17] setup at the Paul Scherrer Institute (Fig. 2). It was combined with a Johann-type crystal spectrometer, which has been used successfully in high-accuracy x-ray measurements involving exotic atoms [24,25]. The ion source, owing to its high mirror ratio, efficiently confines electrons, which results in a large number of highly charged ions being produced [26]. ECR ion sources have two main advantages compared to electron beam ion traps. First, they are a much brighter source of x rays and are, therefore, suitable for obtaining high-statistics spectra in a short period of time; we have recorded as many as 50 000 x rays per hour in a single line. The second advantage is a quite small Doppler effect on transitions because of the low ion velocities (of the order of 15–30 meV in Ar, for a kinetic energy of 1–5 eV) [27,28]. Furthermore, the broadening is symmetrical, so its influence on the position of spectral lines is negligible. The source plasma was excited with a microwave power of typically 200 to 400 W at 6.4 GHz. Its size was found to be of the order of a few centimeters in diameter. For the x-ray measurements, collimators of 28 mm (horizontal) $\times$ 1–4 mm (vertical) were installed in order to reduce background and to prevent an overillumination of the detector.

X rays were diffracted by spherically bent single crystals and recorded with an array of charge-coupled devices (CCDs) positioned near the x-ray focus. This crystal both focuses and disperses x rays. The energy resolution of the spectrometer setup was determined experimentally from the narrow $M1$ lines and was found to be between 0.3 and 0.5 eV [17], depending on the crystal and aperture applied. This is only about 10% larger than the theoretical limit obtained from the dynamical theory of diffraction and, hence, partly superior to other setups [18]. The dominant part of this additional broadening—modeled by a Gaussian—is attributed to imperfections in the crystal setup, because it was found to depend on crystal aperture and material only but not on the target gas or target condition.

Several Bragg crystals were used that were made of silicon or natural quartz and cut along various lattice planes. Having a large area (7.5 and 10 cm in diameter), x rays could be collected with high efficiency. The crystal planes were bent spherically to a radius of about 3 m using a specially developed technique. The quality of their surface and bending was checked with "zebra pattern" tests [29]. Various apertures in front of the crystals limited the reflection area allowing a systematic study of the geometrical line broadening.

A spectrometer setup in Johann geometry, here with a spherically bent crystal, enabled the simultanuous measurement of an energy interval corresponding to the extension of x-ray source and detector along the direction of dispersion. Using as detector a 2×3 matrix of adjacent high-efficiency, low-noise CCDs of 24×24 mm^2 with 600×600 pixels each [30], the detector width of 48 mm covered completely the ECRIS collimator opening together with an almost complete overlap with the reflection in the vertical direction. Owing to its granularity, we achieved a full width at half maximum of about seven pixels for the $M1$ line. This detector size allowed the spectrometer to gather most of the diffracted x rays. The CCDs were operated at $-100\,^{\circ}$C using liquid nitrogen so as to minimize dark current noise. Measuring line energies at the ppm level requires a precise knowledge of the detector geometry. Therefore, relative position, rotation angle, and pixel size of each of the CCDs were measured in a dedicated experiment [31].

The spectral lines as seen on the CCD were vertical and slightly curved because of the imaging properties of the bent crystal. In order to perform the fit procedure using a 1D curve, we straightened the whole pattern and projected it along the axis of dispersion. The curvature was determined by doing a parabolic fit on one of the strong spectral lines and removed by shifting pixel coordinates according to the parabola parameters (Fig. 1) [32,33].

III. ANALYSIS

All line energies were determined by comparing the projected experimental spectra with spectra obtained through simulations. Thus, the whole apparatus was simulated, along with both the $M1$ reference transition and the line under study. The simulated two-dimensional distributions of x-ray hits in the detector plane were then straightened with the same parabola parameters as obtained from the measured spectra. The energy of the line of interest was determined by finding out for which energy the simulated one-dimensional spectral line was best superimposed on the experimental one. This is in contrast to the usual method of determining the energy corresponding to a line by measuring its centroid or peak position and converting it to an energy through some approximate dispersion relation. Our procedure has the advantage of correctly handling the crystal and spectrometer response functions; it thus fully includes any line asymmetries.

The simulation of each spectral line involved modeling the ion source, the crystal, the apertures, and the CCD. The trajectories of x rays in this complex system were calculated with a custom Monte-Carlo ray-tracing code. It turned out that taking into account the intensity distribution in the source along the dispersion direction was essential for obtaining fully consistent line energies from different setups. Hence, the intensity distribution was included in the simulation as measured using a scan of the source performed by a simultaneous rotation of the crystal and of the detector arm by the same angle. The diffraction of x rays by the crystal uses a crystal response function calculated with XCrystal (included in the package XOP [34]) taking into account the temperature of the individual measurements. The measured crystal asphericity [15] and asymmetry angle [35] were included in the simulation. The x rays simulated in this way were then imaged on a virtual CCD. Crystal imperfections were included by convolution with the abovementioned Gaussian contribution [17].

The uncertainty on most of the measured energies comes in equal parts from statistical and systematic errors. The main observed sources of systematic error were intensity distribution in the ion source, the crystal curvature radius (±0.5 mm), the crystal-CCD distance (±0.25 mm), the centering of the various mechanical elements, and, in the case of quartz, the lattice spacing. In order to identify additional sources of systematic errors, many spectra were acquired under different experimental conditions, and energies were checked for consistency.

During a run with a single element (S, Cl, or Ar), we recorded the same spectra using several crystal setups: (i) different crystal materials (quartz or silicon), (ii) different crystals of the same type, (iii) crystals cut along different planes, and (iv) different apertures in front of the crystal [32,33]. In addition, the detector was positioned at various locations in the vicinity of the calculated x-ray focus. The consistency of the results is shown for the case of quartz (100) crystals in Fig. 3.

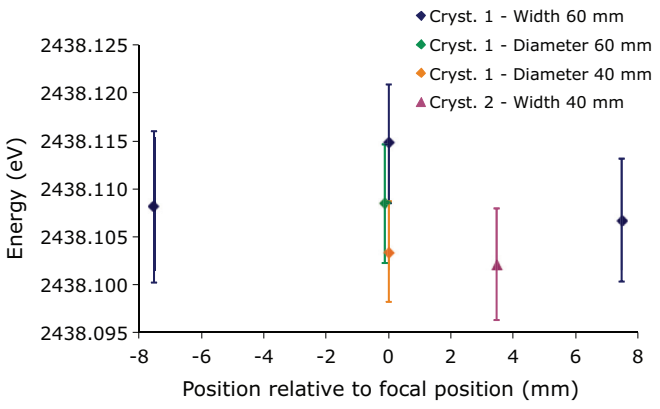

FIG. 3. (Color online) Measurements of the $1s\,2s\,2p\ ^2P_{3/2} \rightarrow 1s^2\,2s\ ^2S_{1/2}$ line in three-electron sulfur. Internal consistency checks were performed by recording spectra for various distances between the Bragg crystal and the x-ray detector using two different (100) quartz crystals (represented here using different symbols) and four different apertures (represented by different colors). The apertures leave apparent a circle or a band of the crystal, their size being given in the legend.

We also searched for possible line contamination by satellite transitions. First, we examined calculations of ion production in the plasma [36,37] in order to assess whether potential satellite lines could contaminate the measured ones; these satellite lines appear to be either far enough in energy or too weak (compared to statistical uncertainties in the experimental spectra) to affect our energy determination. Secondly, the residues of all line fits were plotted, and no hint for satellite contamination was found.

IV. RESULTS

All the energies given in Table I that have been previously measured are consistent with the ones reported in the literature; we were thus able to check all the transitions for argon [38–41], and some of the transitions for sulfur [42,43]. Six of the reported energies have never been measured before (see Table I).

Except for $1s\,2p\ ^3P_2 \rightarrow 1s^2$ in sulfur [43], the energies in Table I improve the existing experimental accuracy by a factor of about 3 to 50. As an additional check, we measured the $1s\,2s\ ^1P_1 \rightarrow 1s^2$ transition energy in Ar and found 3139.567(11) eV. This result is at about one standard deviation from the value of Ref. [18] and, therefore, is regarded to be consistent with it.

The energies in Table I were also compared to theoretical values. Of particular importance are the values from the work of Artemyev *et al.* on 2-electron ions [21], since it is where the energies of our reference M1 transition were taken from: values are within one standard deviation of our experimental results, except for $1s\,2p\ ^3P_1 \rightarrow 1s^2$ in Ar (see Table II). For this excited level, the theory prediction deviates about 3 standard deviations from our value, which is, however, compatible with Deslattes' accurate measurement [40]. We also compare our values to very accurate uv measurements of the $1s\,2p\ ^3P_2 \rightarrow 1s2s\ ^3S_1$ transition energy in sulfur [43] and in argon [44–46] in Table II. The agreement between our values and the uv measurements is excellent.

Experimental accuracies achieved for two-electron ions are about the magnitude of two-photon QED effects between electrons [21]. For instance, the total two-electron QED corrections for the $1s\,2s\ ^3S_1$ and the $1s\,2p\ ^3P_1$ levels in He-like Ar are −0.0148(1) eV and −0.0031(1) eV, respectively, giving an energy difference of approximately our uncertainties (see Table I or II). For three- and four-electron ion transitions, theoretical values typically deviate by 20–50 meV from our results (except 300 meV for the Be-like line in argon) [47–51]; which is similar to the typical scattering among the theoretical results themselves (which are given without any uncertainty). Thus, except for the $1s\,2p\ ^3P_1 \rightarrow 1s^2$ transition in Ar [21], our experimental energies are consistent with published calculated values.

V. CONCLUSIONS

In summary, six new x-ray line transitions were measured, and the accuracy on six other line energies was improved. They are consistent between different experimental setups, and also with existing theoretical and experimental results (with one exception). The measurements were made with a very-well-characterized spectrometer. The data analysis was performed

TABLE II. Transition energies (in electron volts) of two-electron sulfur and argon. The energies of the $M1$ reference transitions used in this work were taken from Ref. [21].

Element	Transition	This work	Theory [21]	Experiment	
S	$1s\,2p\ ^3P_2 \rightarrow 1s^2\ ^1S_0$	2448.739 (20)	2448.7628(3)	2448.7621(9)	[43]
	$1s\,2p\ ^3P_1 \rightarrow 1s^2\ ^1S_0$	2447.15(9)	2447.1439(3)	2447.05(11)	[42]
	$1s\,2p\ ^3P_2 \rightarrow 1s2s\ ^3S_1$	18.388(20)	18.4116(4)	18.4108(6)	[43]
Ar	$1s\,2p\ ^1P_1 \rightarrow 1s^2\ ^1S_0$	3139.567(11)	3139.5821(4)	3139.583(6)	[18]
	$1s\,2p\ ^3P_2 \rightarrow 1s^2\ ^1S_0$	3126.291(11)	3126.2896(4)	3126.37(24)	[41]
				3126.282(36)	[40]
	$1s\,2p\ ^3P_1 \rightarrow 1s^2\ ^1S_0$	3123.489(12)	3123.5344(4)	3123.53(24)	[41]
				3123.520(36)	[40]
	$1s\,2p\ ^3P_2 \rightarrow 1s2s\ ^3S_1$	22.143(11)	22.1413(4)	22.1423(6)	[44]
				22.142(4)	[45]
				22.13(4)	[46]

using a dedicated x-ray tracing simulation embedding all the relevant physics. The absolute values we report here for the energy transitions are calibrated using the precise theoretical prediction of the $M1$ line energies which served as references (shifting the $M1$ energies would shift our values).

Many of these energies only have a few-parts-per-million uncertainty, which puts them among the most precise spectroscopy results in highly charged ions [18,22,52,53]. This accuracy makes the measured energies sensitive to two-photon QED effects, mostly through the $1s\,2s\ ^3S_1$ level [21].

The lines reported in this article were measured for the sequence of nuclear charge numbers $Z = 16$–18. Line energies can be used for comparison with the theoretically predicted Z dependence and, more generally, for testing results of atomic calculation approaches. For three- and four-electron transitions, however, where theoretical results for line energies are widely scattered, a meaningful comparison given the achieved experimental accuracy requires first the calculation of Auger shifts.

The multiple lines that we measured for each element (see Fig. 1) significantly increase the number of available transition energy standards in the 3-keV region [13,18,22,54]. Their energies can thus prove very useful for spectroscopic line identification. The lines in Table I can furthermore be used in relative energy measurements (for instance, with exotic atoms [29], in astrophysics [55,56], at synchrotron facilities, or with free-electron lasers [57]) or for calibration (e.g., the characterization of x-ray microcalorimeters [58]).

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